Development of Combinatorial Methods for Alloy Design and Optimization

(CPS# 1778)

~ IMF Knowledge Base & Core Activities Program ~

Participants:

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National Lab: Easo P. George & Michael L. Santella, Oak Ridge National Laboratory, Metals & Ceramics Division

Industry: Duraloy Technologies

PROJECT SUMMARY

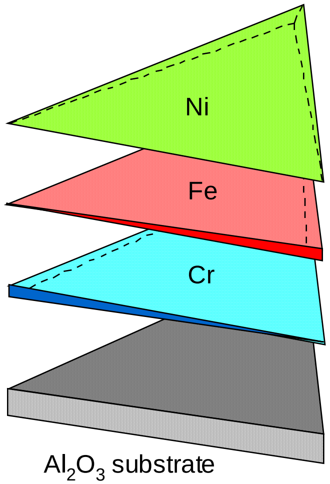
Development of Combinatorial Methods for Alloy Design and Optimization (CPS# 1778)

Goal: Develop a comprehensive methodology for designing and optimizing metallic alloys by combinatorial principles.

Challenge: Significantly reduce the time and expense needed for alloy design.

Benefits: Expedite alloy design process; reduce alloy design costs; explore larger number of alloy compositions; identify and optimize new alloys with improved properties for energy savings

FY05 Activities: Complete measurement of alloy library mechanical properties by nanoindentation; assess carburization performance and corrosion resistance of alloy libraries; measure properties of conventionally prepared bulk alloys for comparison to libraries; complete study of alloying by e-beam welding techniques; prepare final report



Schematic of thin film alloy preparation

Participants: University of Tennessee, Oak Ridge National Laboratory; Duraloy Technologies

BARRIER-PATHWAY APPROACH

Development of Combinatorial Methods for Alloy Design and Optimization (CPS# 1778)

Barriers

- Lack of techniques for producing large quantities of alloys quickly and inexpensively
- Lack of techniques for rapidly screening the structure and properties of alloys

Pathways

- Develop techniques for producing "alloy libraries" based on thin film deposition and processing
- Develop techniques for rapid structure and property characterization (synchrotron x-ray analysis, nanoindentation, carburization, corrosion, etc.)

Critical Metrics

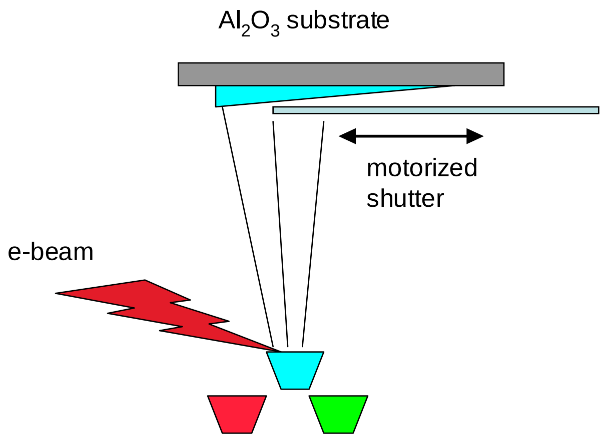
- Apply methodologies to the Fe-Ni-Cr ternary alloy system; compare to properties of conventionally prepared alloys

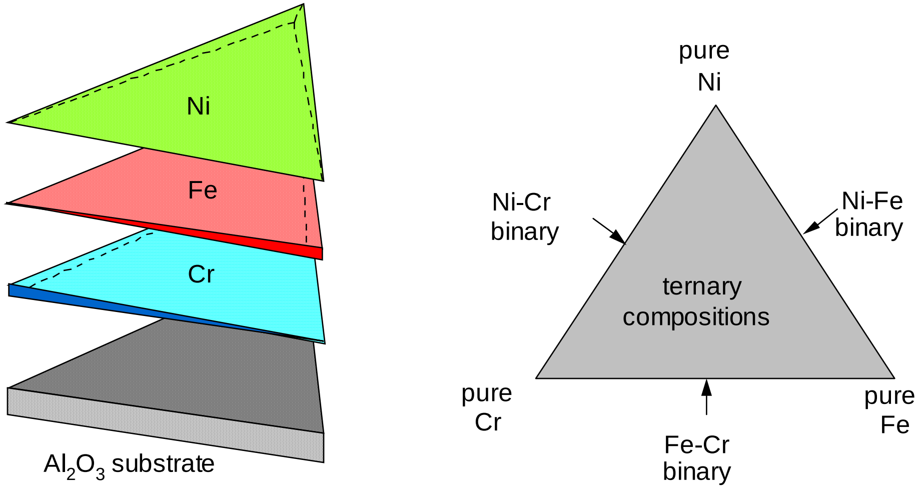
Benefits (est.)*	2020
Energy Savings	38 trillion BTU's
Cost Savings	$185 million
CO_2 Reduction	607 thousand tons
NO_x Reduction	5.3 thousand tons

** calculation assumes 50°C higher use temperature for H-series alloys*

"ALLOY LIBRARY" SPECIMEN PREPARATION

Thin film deposition by shuttered e-beam evaporation



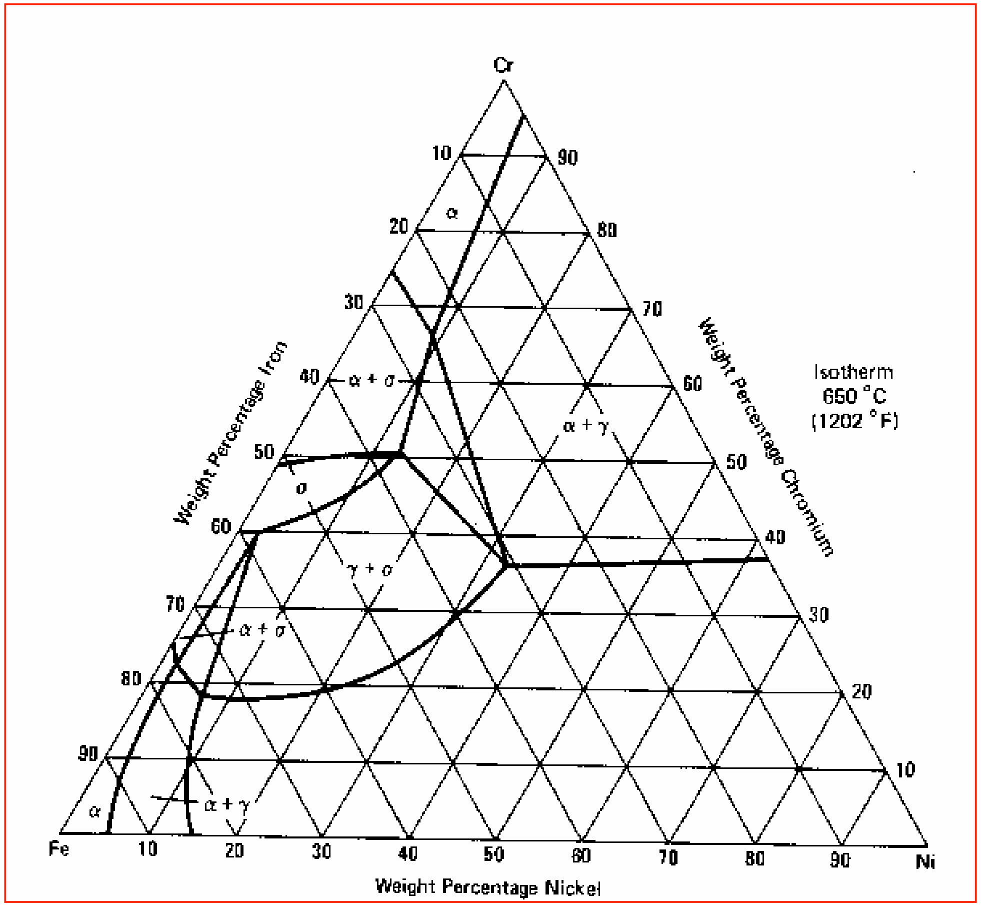


Alloying Techniques:

- *solid state diffusion by annealing*
- *localized heating and melting:*
  - *- focused e-beam welder*
  - *- laser (Nd:YAG)*

MODEL ALLOY SYSTEM: Fe-Ni-Cr Ternary

- *Foundation for austenitic and ferritic stainless steels*
- *Well studied and characterized*
- *Includes H- and C-series heat and corrosion resistant casting alloys of interest to industrial partners*



PROGRESS: YEAR 1
(April 1, 2002 - March 31, 2003)

- *Designed and constructed controllable shutter system for the electron beam vapor deposition system*
- *Developed procedures for depositing wedge profiled films of iron, chromium, and nickel on sapphire substrates*
- *Determined optimum annealing conditions for alloying by solid-state diffusion*
- *Examined quality of the first ternary alloy library by synchrotron x-ray diffraction and fluorescence (APS at ANL)*
- *Began to explore alternative methods for alloying by electron beam, laser, & IR melting*

Graduate student making thin films in e-beam deposition system

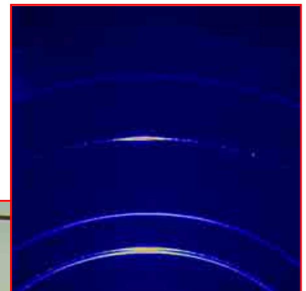
PROGRESS: YEAR 2
(April 1, 2003 - March 31, 2004)

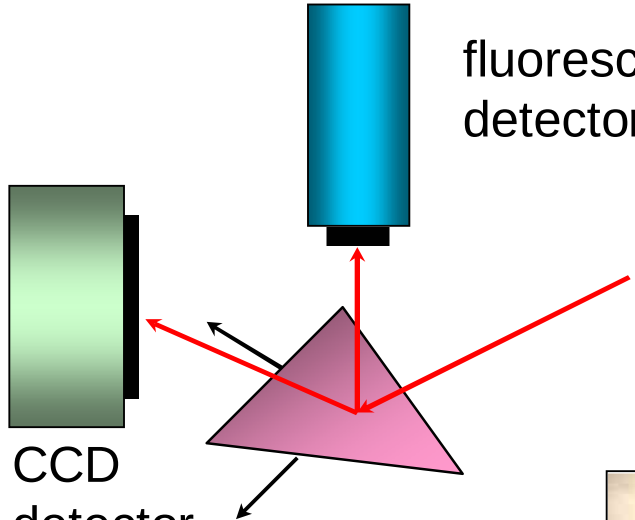
- *Completed alloy library preparation by thin film deposition and solid state diffusion (techniques developed to minimize oxide formation)*
- *Completed development of synchrotron-based techniques for rapid structural and chemical characterization*
- *Developed new technique for alloy library preparation by co-sputtering from three separate magnetron sources*
- *Began measurement of mechanical properties by nanoindentation*
- *Developed electron beam melting techniques as an alternative for thin film alloying*
- *Prepared bulk Fe-Ni-Cr alloys by arc-melting and casting for comparison to alloy libraries*

Electron beam melting system at ORNL

RAPID STRUCTURAL ASSESSMENT BY SYNCHROTRON TECHNIQUES

Advanced Photon Source at Argonne National Lab

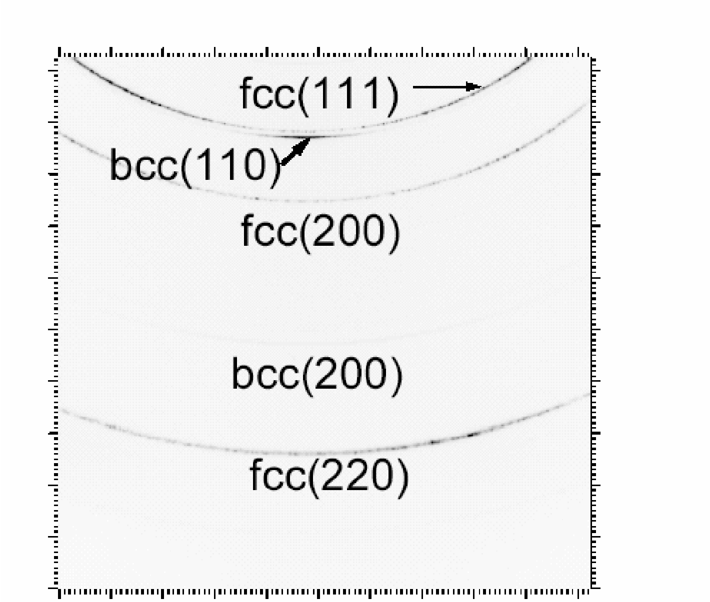


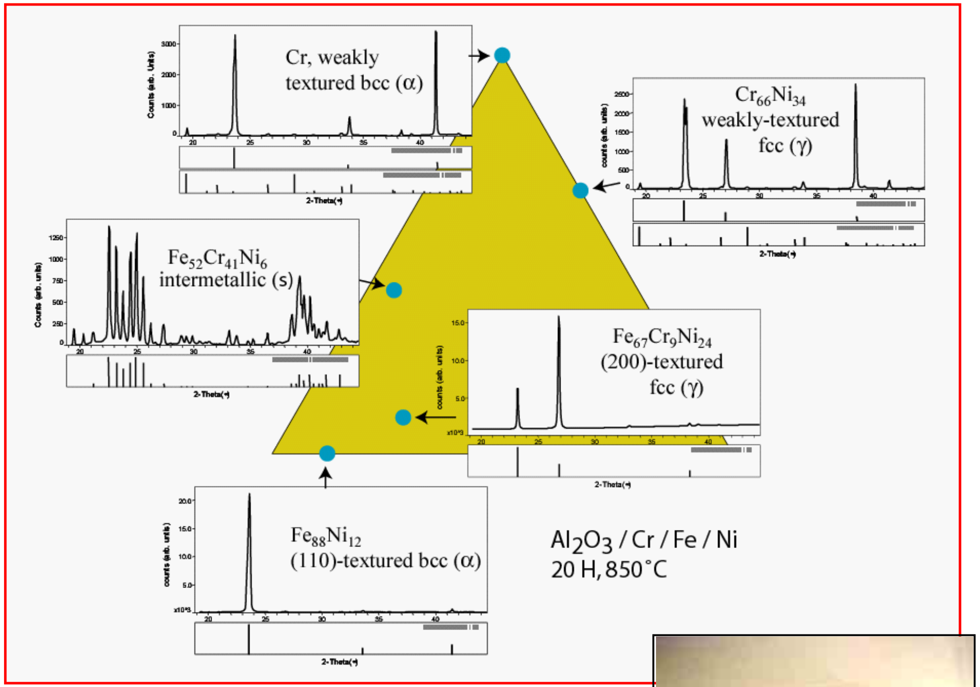


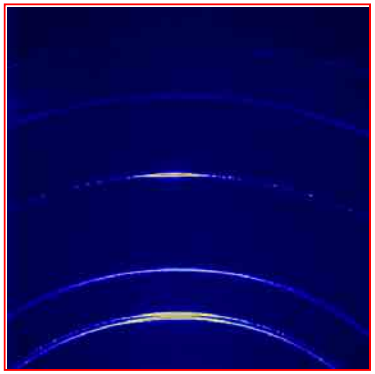
- *x-ray diffraction and fluorescence measured from 2600 positions in 4 hours*

SYNCHROTRON MEASUREMENT CAPABILITIES

- *phase identification*
- *crystal structure*
- *lattice parameters*
- *texture and orientation*
- *grain size*
- *chemical composition*

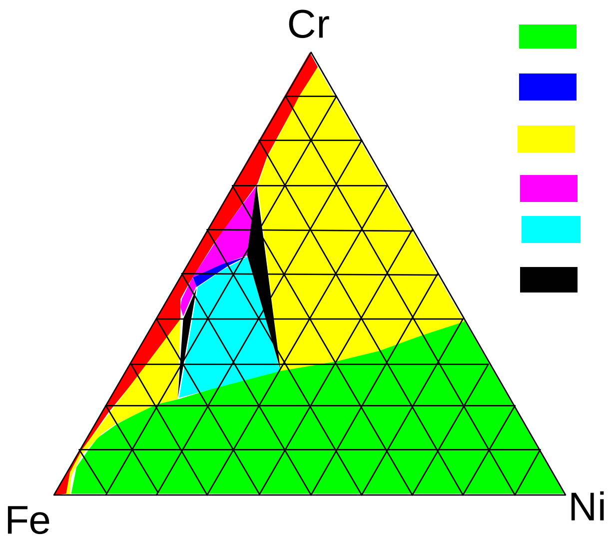






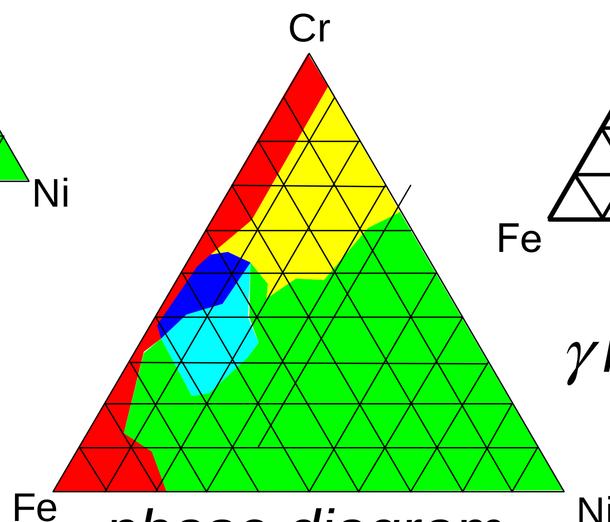
PHASES & LATTICE PARAMETERS

- α(bcc)
- γ(fcc)
- σ(tetragonal intermetallic)
- $\alpha + \gamma$
- $\alpha + \sigma$
- $\gamma + \sigma$
- $\alpha + \gamma + \sigma$

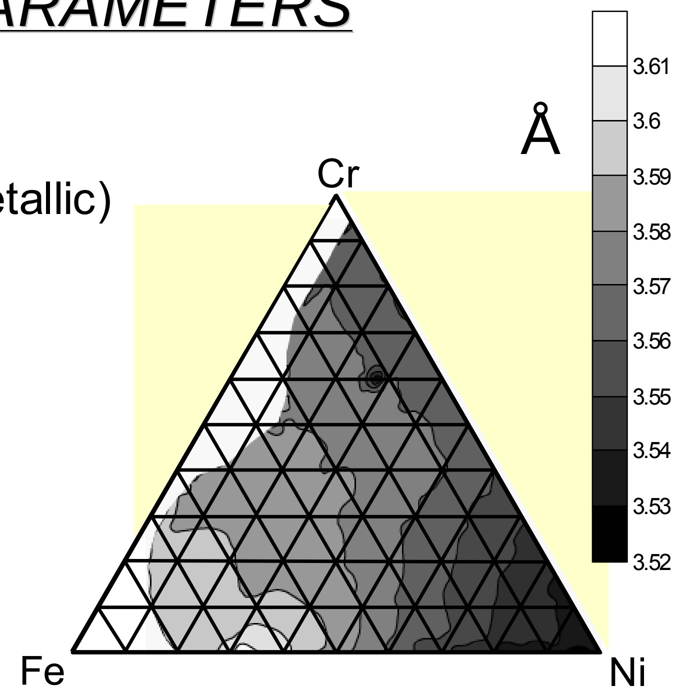


phase diagram from handbook

T = 850°C



phase diagram as measured

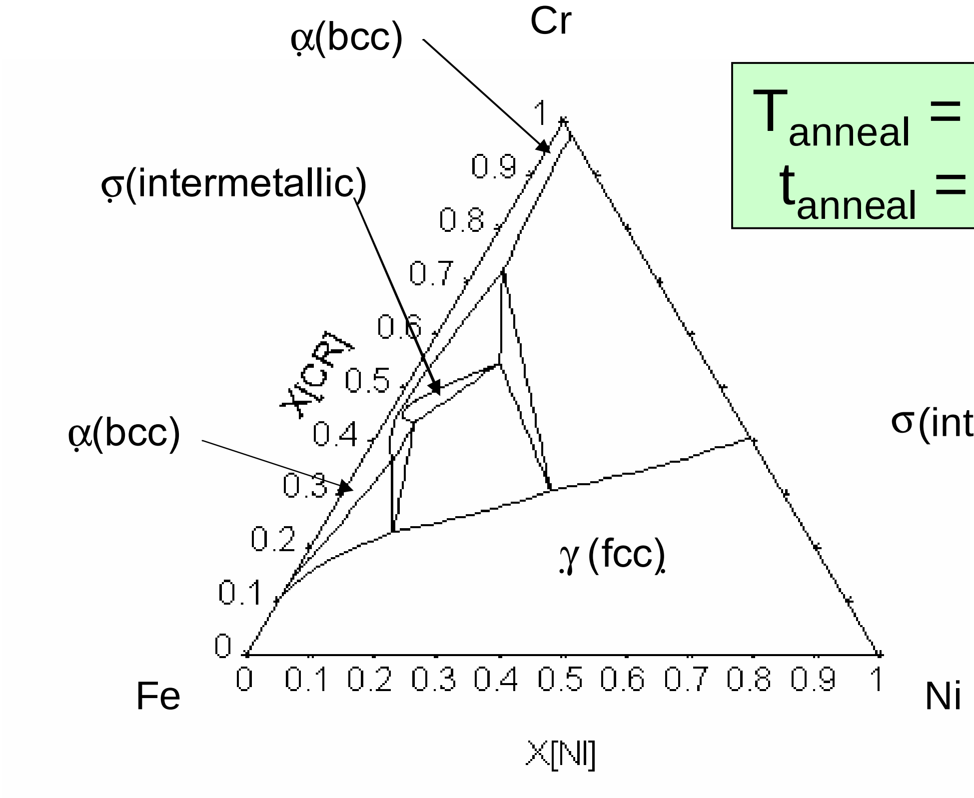


γ lattice parameters

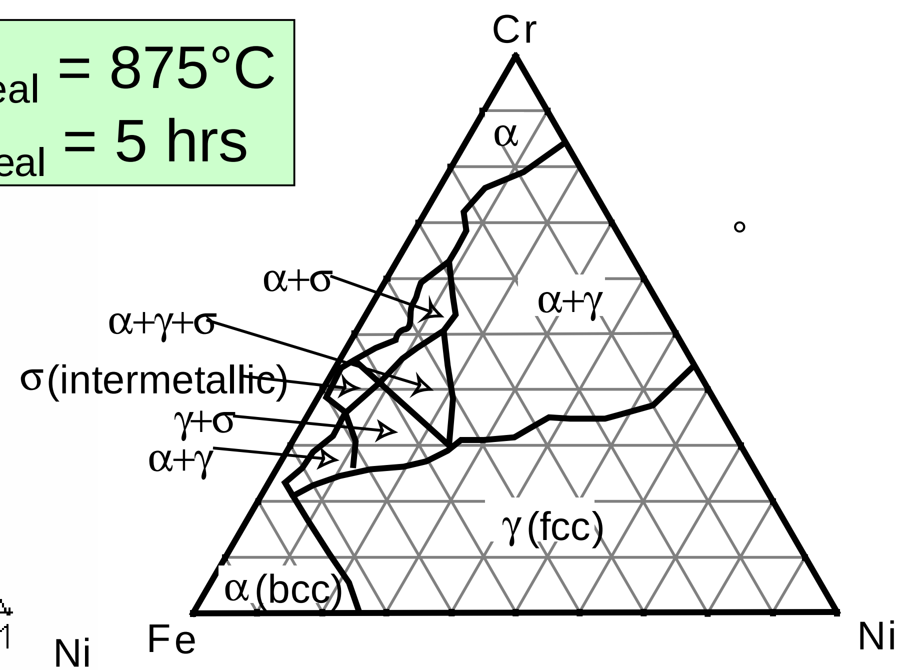
- paper published, *Journal of Materials Research (2003)*

OUR "BEST" ALLOY LIBRARY

(optimal annealing conditions and minimum oxide formation)



T_{anneal} = 875°C
t_{anneal} = 5 hrs

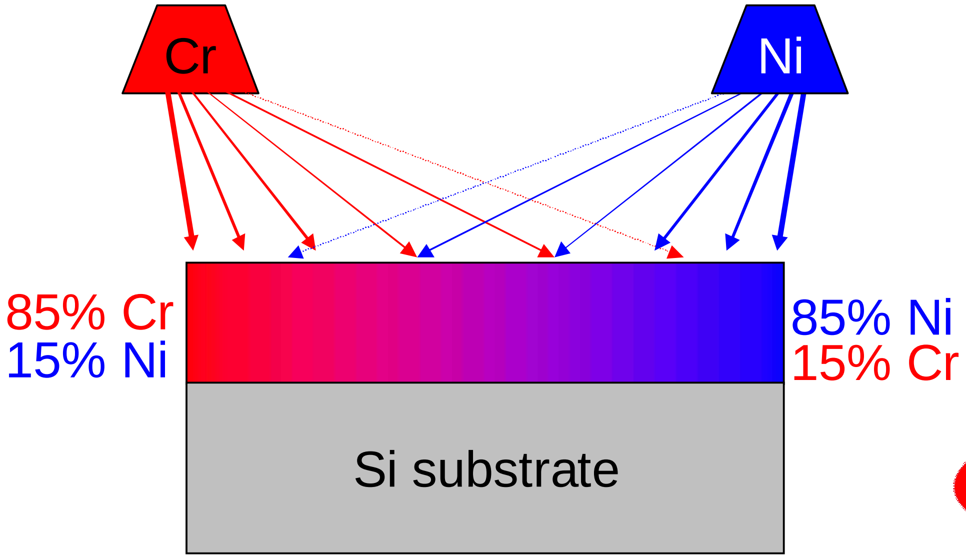


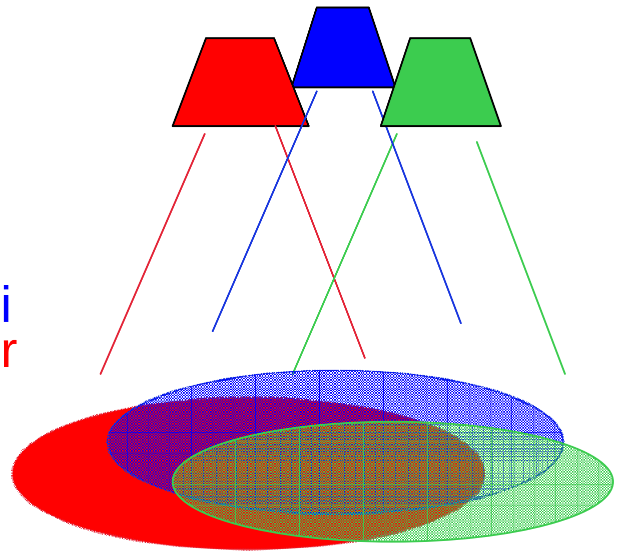
conventional equilibrium diagram

phase diagram as measured

CODEPOSITION BY MAGNETRON SPUTTERING

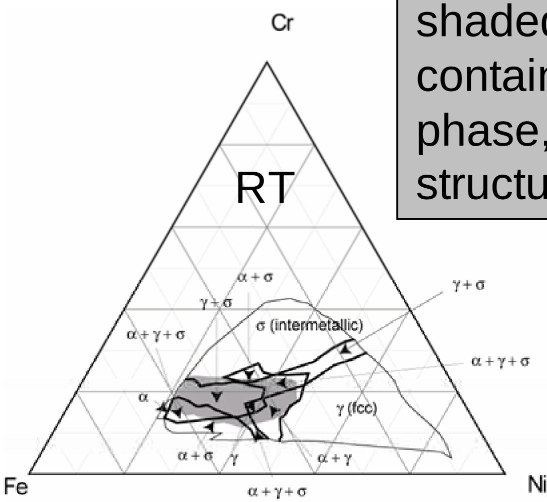
binary systems *ternary systems*



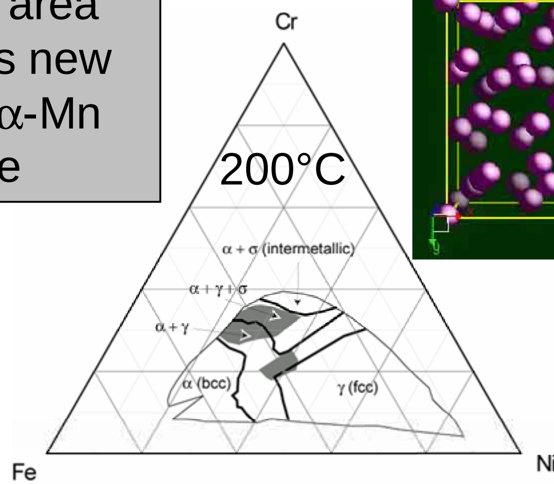


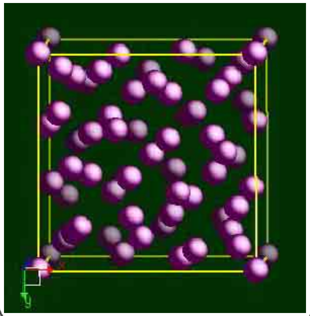
- *Advantage:* no alloying required - mixing occurs during film deposition
- *Disadvantage: cannot cover full range of composition*

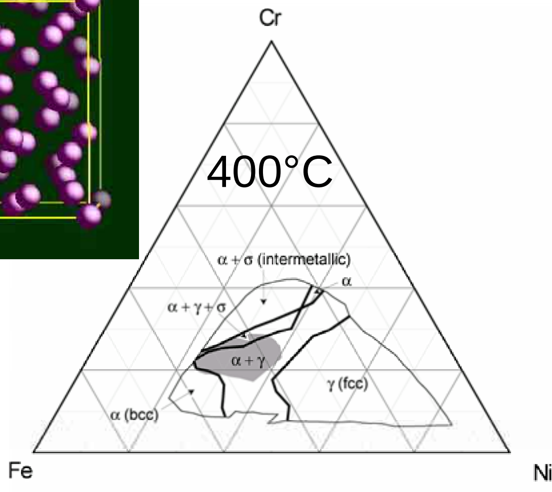
INFLUENCE OF ANNEALING AFTER DEPOSTION

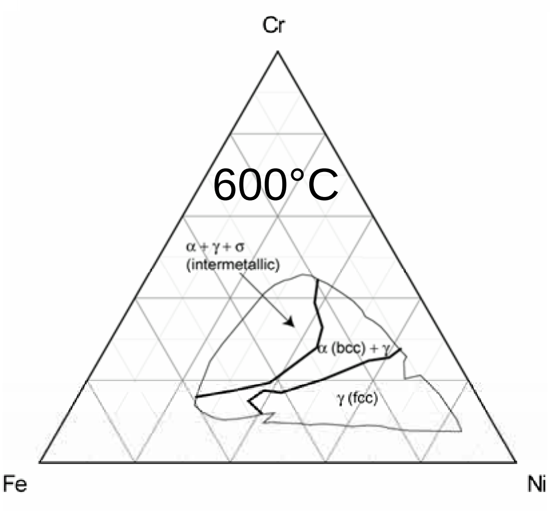


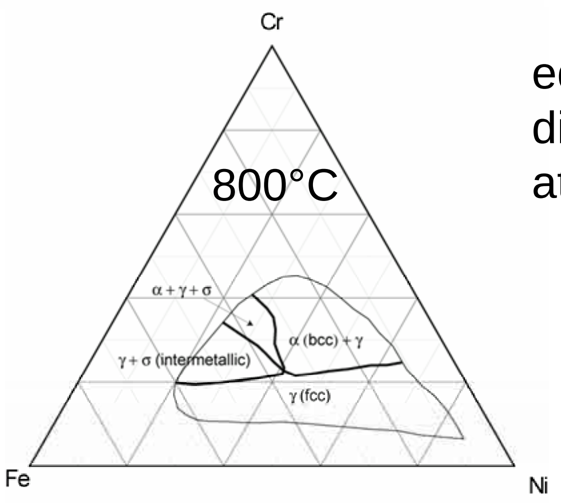
shaded area contains new phase, α-Mn structure



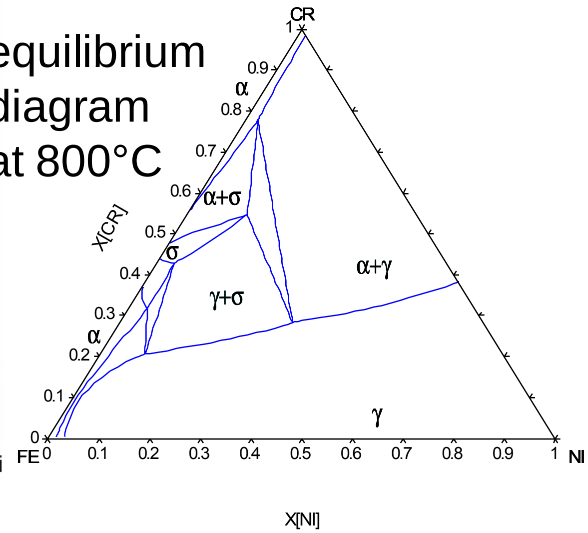




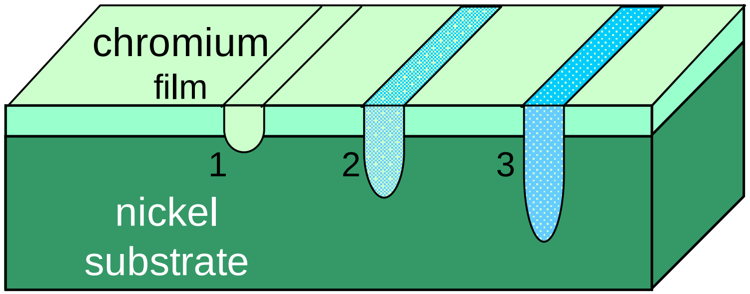


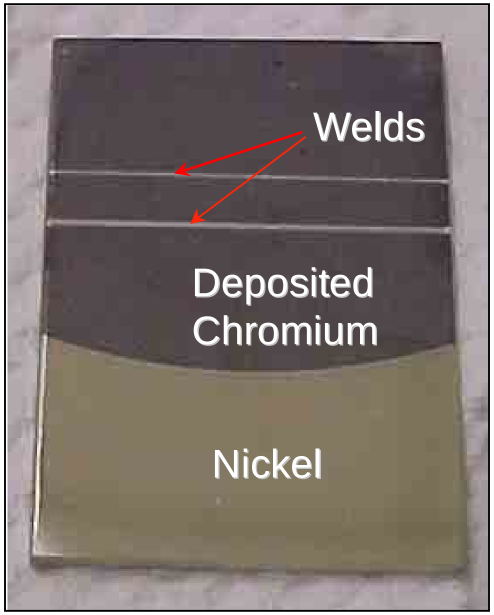


equilibrium diagram at 800°C



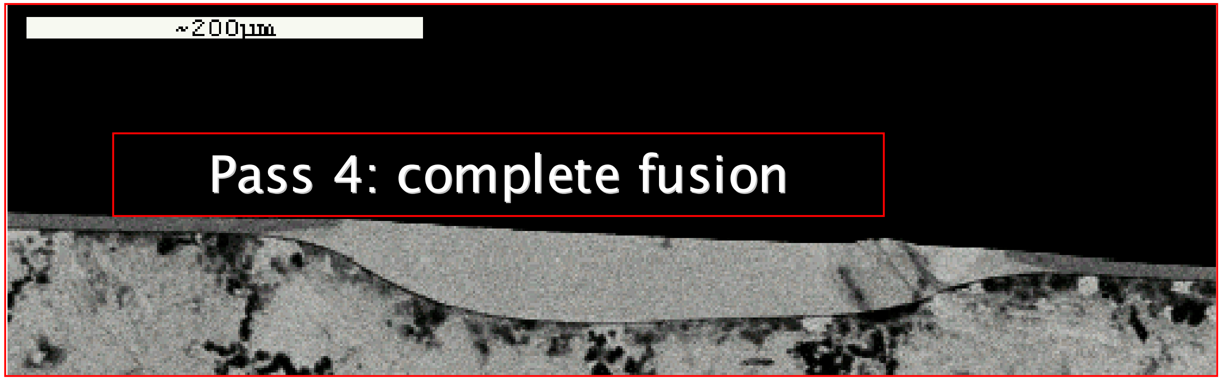
FOCUSED ELECTRON BEAM MELTING: Cr on Ni

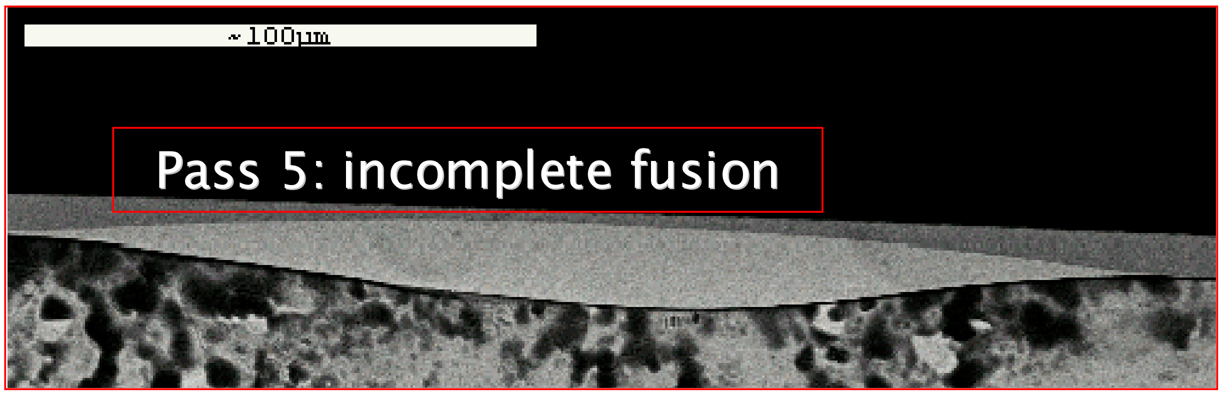




	Voltage (V)	Current (mamps)	Speed (in/min)
Pass 1*	75	0.2	50
Pass 2	75	0.5	50
Pass 3	75	1.0	50
Pass 4	75	1.5	50
Pass 5	150	0.7	50
Pass 6*	150	0.3	20
Pass 7*	150	0.3	5
*no marking visible on surface after weld			

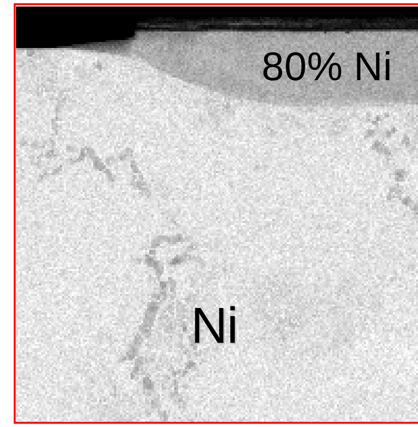
e-BEAM WELD PASSES IN CROSS SECTION

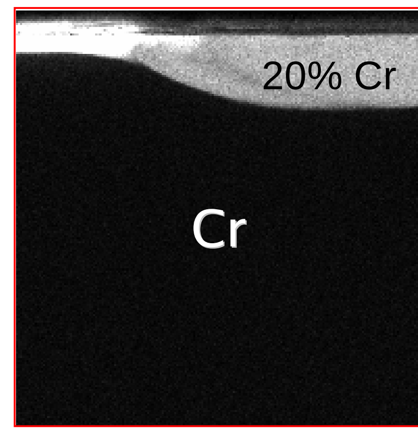




- *composition very uniform in fusion zone*

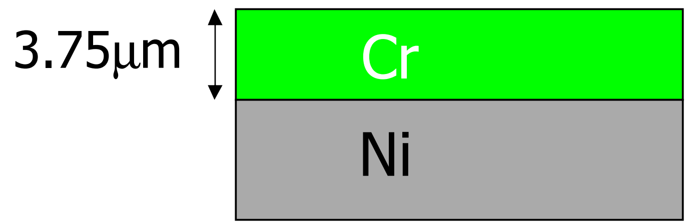
EDS element maps: Pass 4





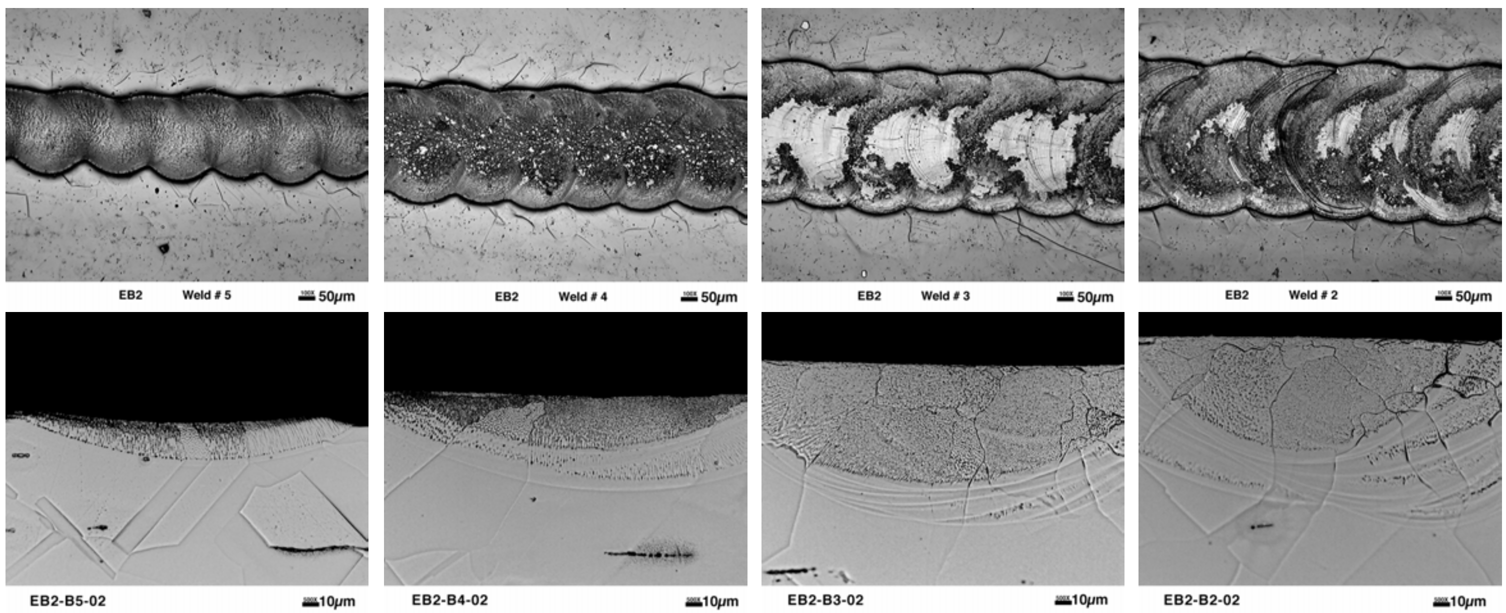
COMPOSITION VARIATION BY CURRENT CONTROL

welding voltage: 150 V
welding speed: 50 in/min



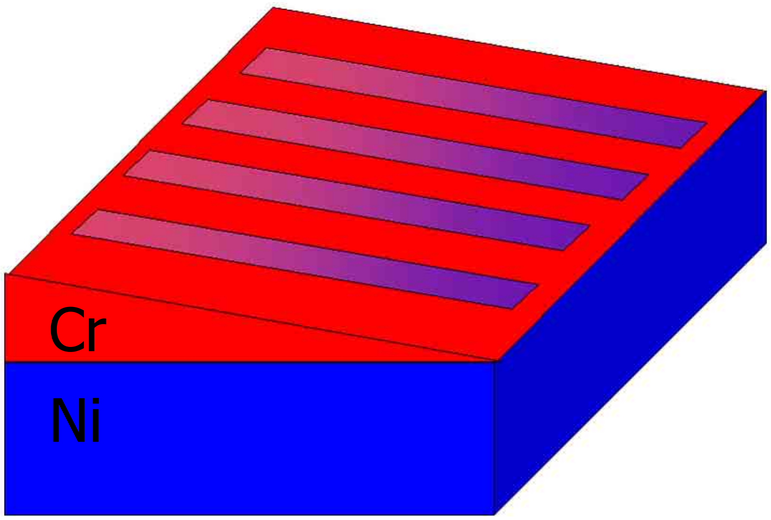
plan view

cross section

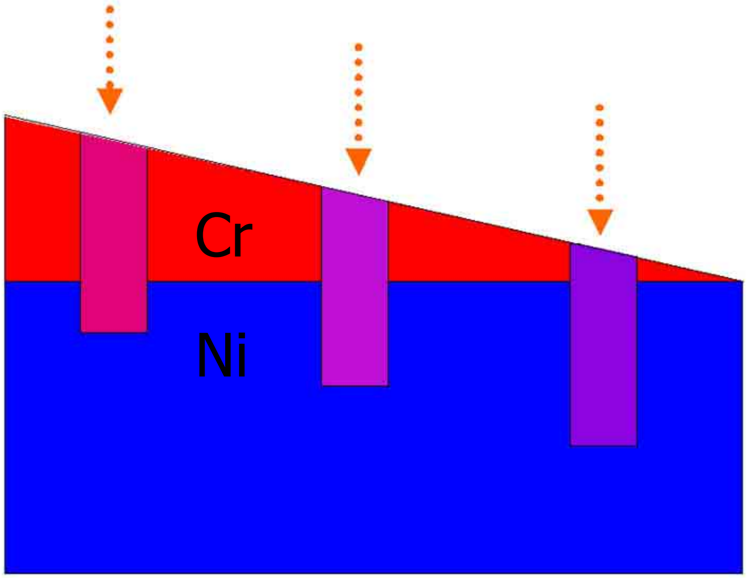


welding current	0.7 mA	0.8 mA	0.9 mA	1.0 mA

COMPOSITION VARIATION BY WEDGING FILMS

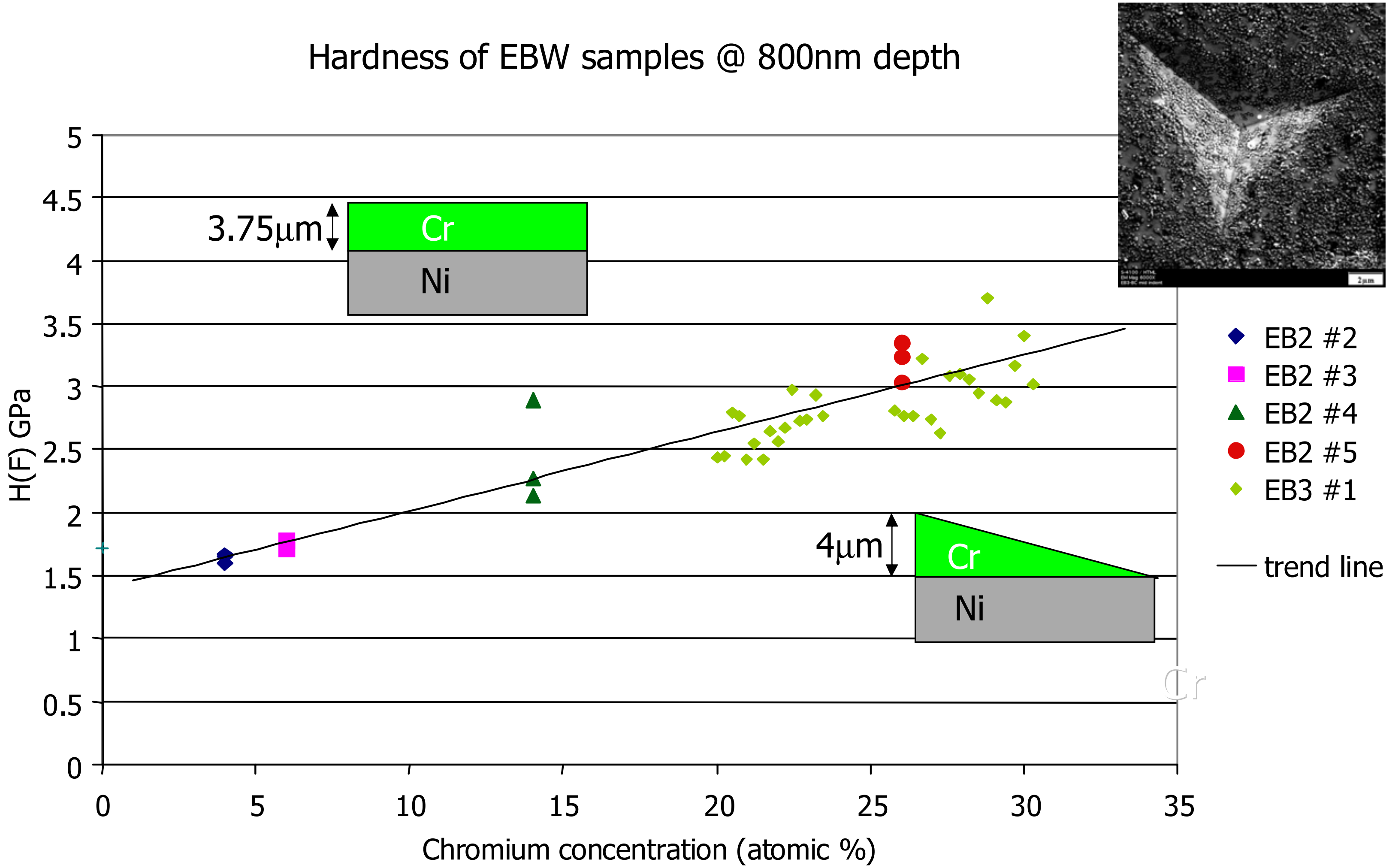


wedged film

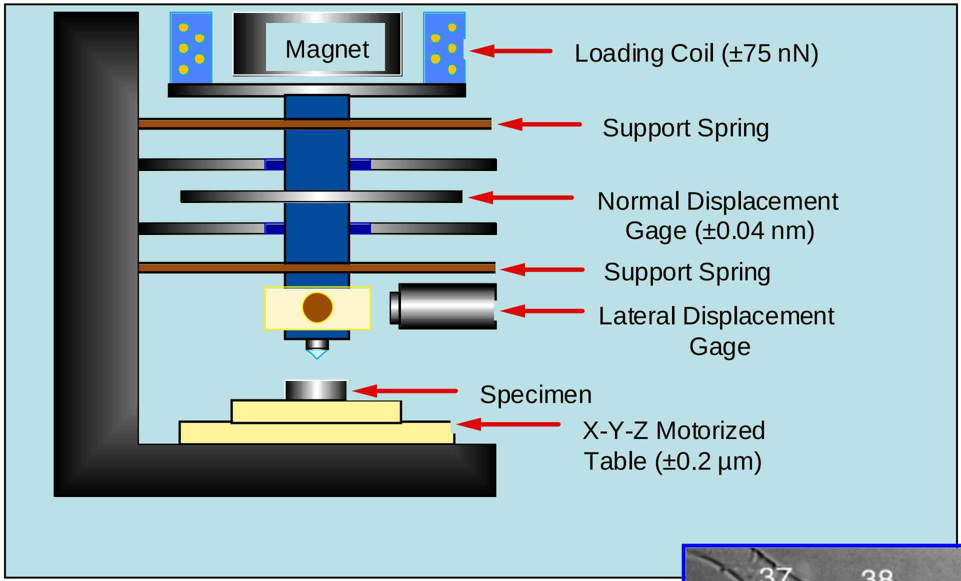


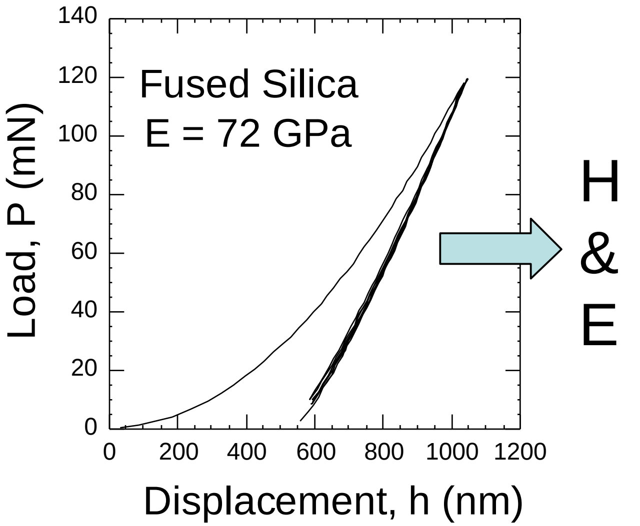
constant current

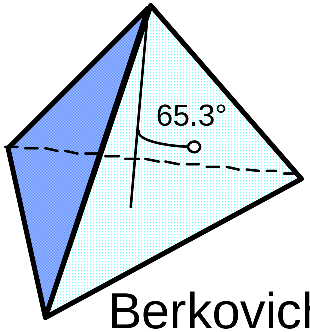
HARDNESS ASSESSMENT BY NANOINDENTATION



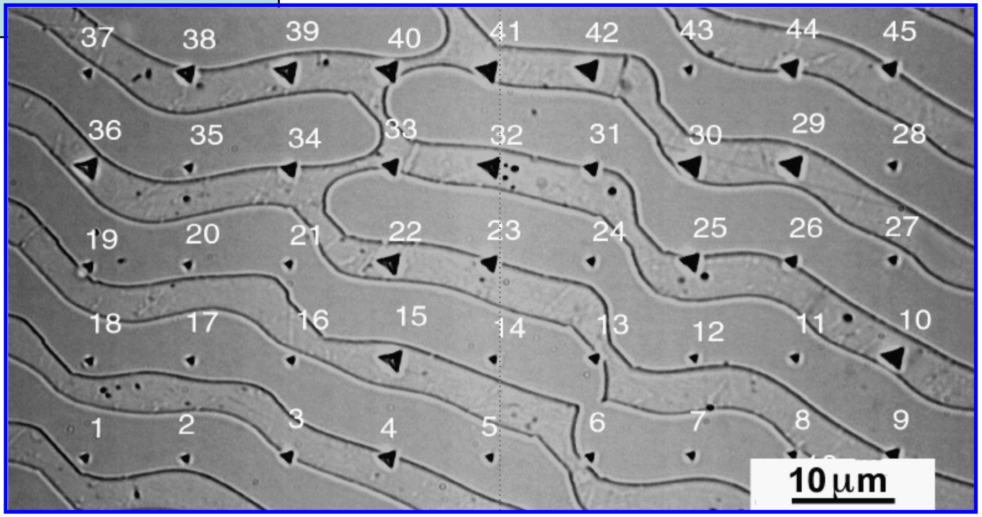
NANOINDENTATION MEASUREMENT OF MECHANICAL PROPERTIES





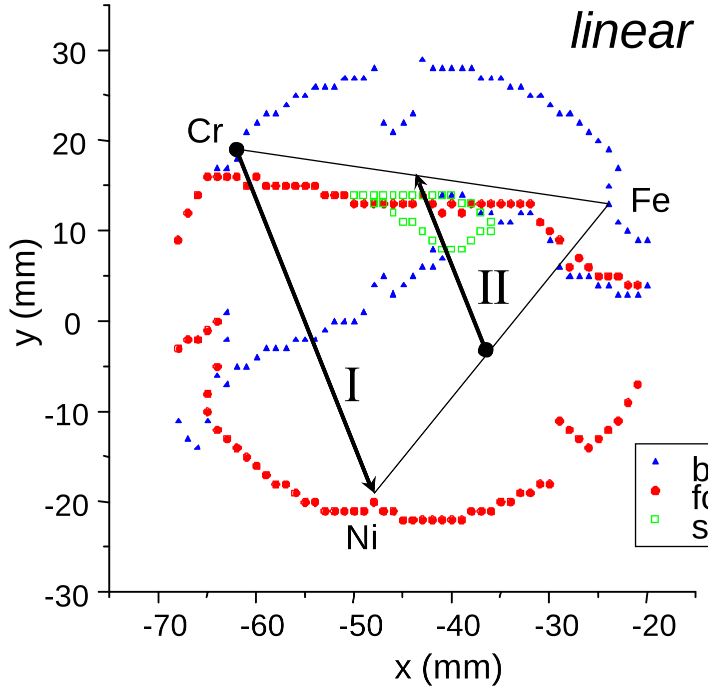


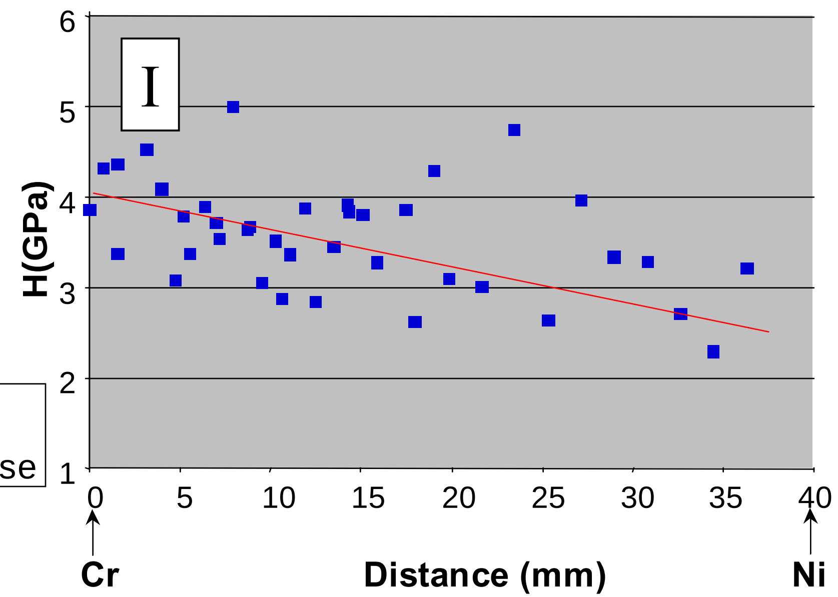
Berkovich indenter

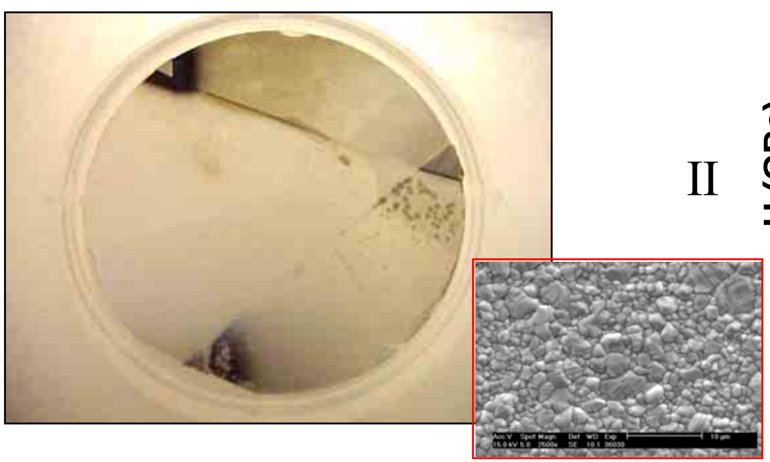


MEASUREMENTS FOR OUR "BEST" LIBRARY

linear hardness traverses







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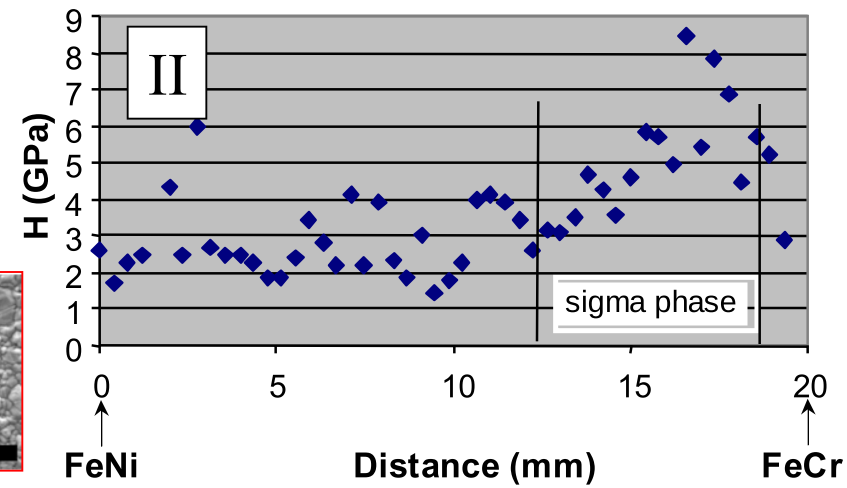
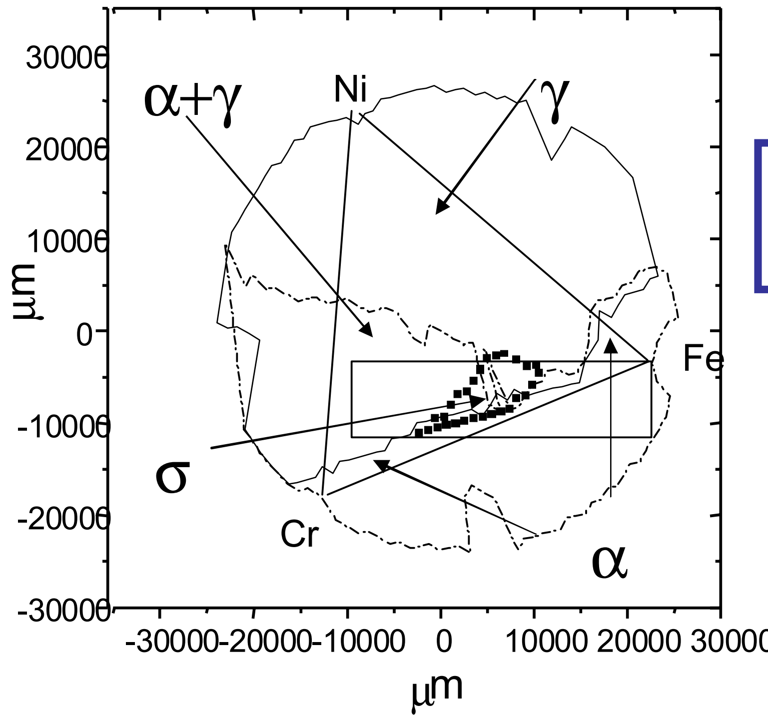
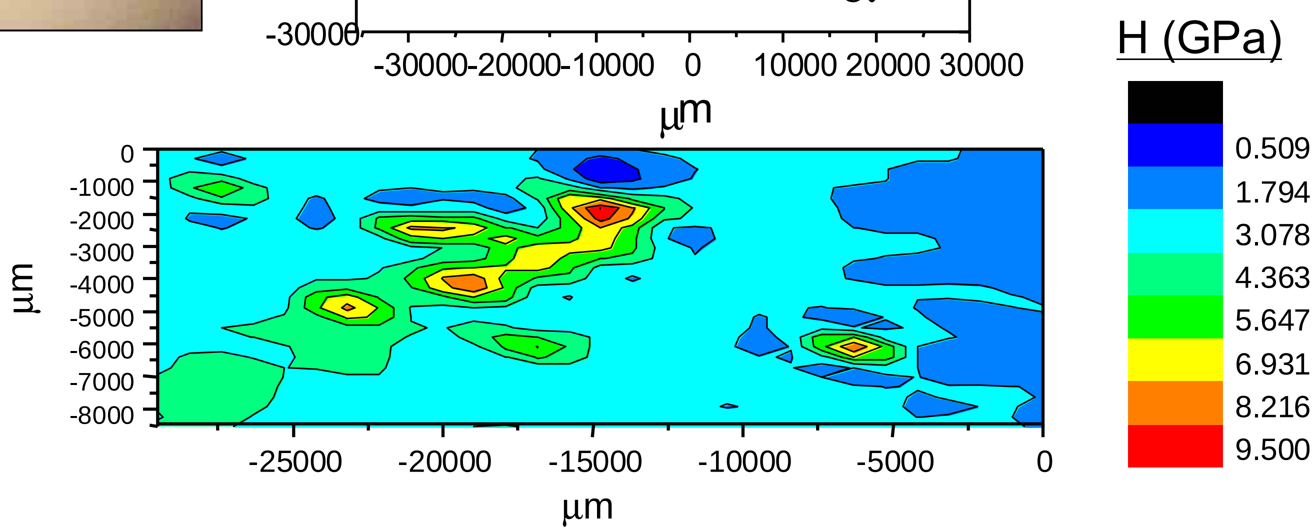


HARDNESS "MAPS"



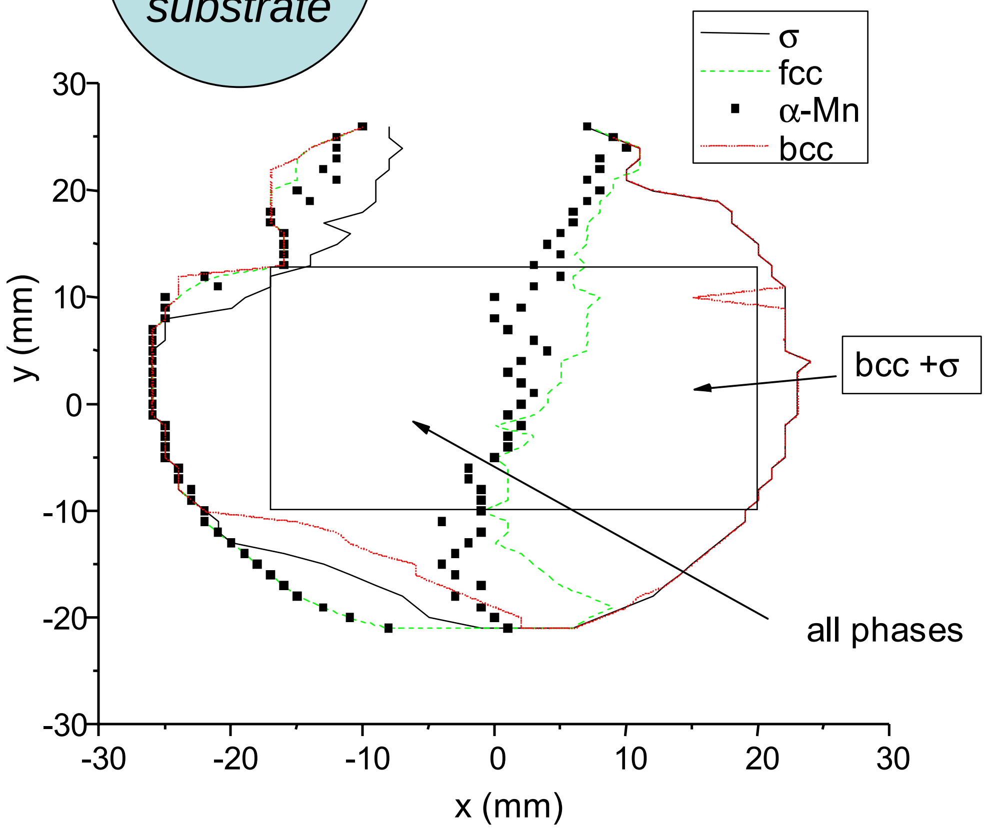
T_{anneal} = 875°C
t_{anneal} = 5 hrs

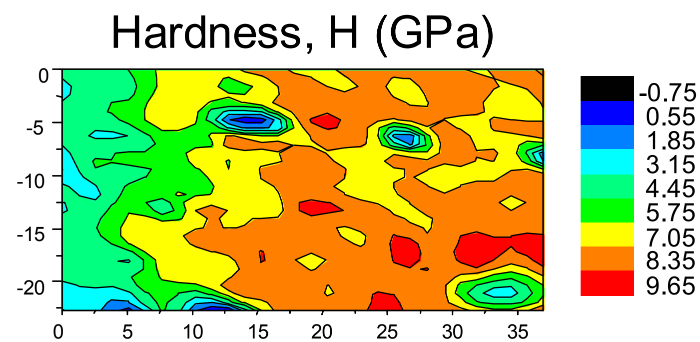


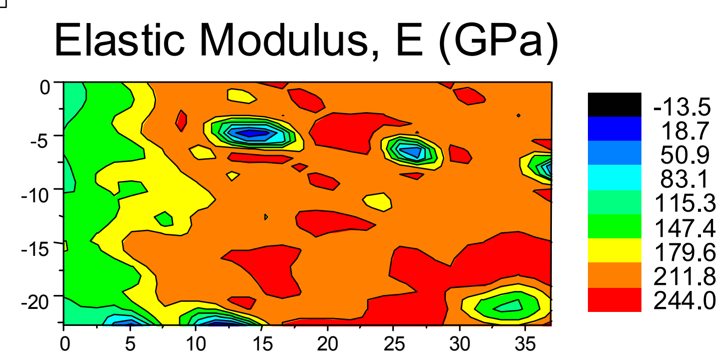
HARDNESS AND MODULUS MAPS

50 mm sapphire substrate

co-deposited Fe-Ni-Cr library; T_{anneal} = 500°C







PLANS FOR YEAR 3

(April 1, 2004 - March 31, 2005)

- *Complete measurement of alloy library mechanical properties by nanoindentation*
- *Assess carburization performance*
- *Assess corrosion resistance*
- *Measure relevant properties of bulk alloys for comparison to alloy libraries*
- *Complete study of alloying by e-beam welding*
- *Prepare final report*